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A vacuum induction furnace for pyrometric cone equivalent determinations

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A VACUUM INDUCTION FURNACE
FOR PYROMETRIC CONE EQUIVALENT DETERMINATIONS

BY

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A

THESIS

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ABSTRACT

Various furnaces which are commonly used for P.C.E. determinations were compared from a literature review. The inadequacies of these furnaces were noted and a furnace was designed, constructed, and evaluated in an effort to minimize or eliminate some of the indicated problems. For this furnace heat is developed in opposed cones (susceptors), which are fabricated from 0.005" molybdenum sheet, by induced (induction) currents from a 10 KW vacuum tube generator. The furnace components are enveloped by a pyrex cylinder in which a vacuum (approximately 1 to 5 micron pressure absolute) is maintained during operation.

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I INTRODUCTION

For many years in the ceramic industry the pyrometric cone has been used to determine the heat treatment that a product undergoes during firing. Advances in science and technology have produced materials which have fusion or deformation temperatures equal to or higher than those of the standard pyrometric cones. Each of the furnaces presently employed to obtain the necessary temperatures or heat treatment has one or more undesirable operating characteristics. It is hoped that this investigation will produce a furnace or will create new ideas in furnace design that will eventually minimize or eliminate the operational problems presently encountered.

Most furnaces currently being used for P.C.E. determinations are limited to cone 37. According to the A.S.T.M.[1] prescribed heating rates, an end temperature of 3308° F (1820° C) is required to deform properly standard cone 37. The useful life of these furnaces, when heated to the temperatures required to deform standard cones 38 through 42, is greatly impaired. In addition to poor life, results are difficult to reproduce in most of these furnaces, and atmospheres are difficult to control. In air-gas and oxygen-gas furnaces excessive gas turbulence causes premature deformation of cones. Hot spots, which develop in electric gran-annular type furnaces, when the gran-annular carbon of the resistance section becomes segregated, produce thermal gradients and erroneous results, and the burning of the carbon produce gases which impair visibility of cones during firing. The above mentioned problems, as well as others, indicate the necessity for a better furnace design.

To date the uses of induction heating techniques have not been extensively used for P.C.E. determinations. The primary reason is to be found in the cost of the radio frequency generator, which has been considered prohibitive for this application. However, the many applications of induction heating, which have been made in the past few years, make this piece of equipment a valuable research tool for any ceramic laboratory. This investigation was undertaken because of this versatility of induction heating in high temperature research.

The purpose of this investigation is to design, construct, and evaluate a furnace utilizing induction heating techniques to minimize or eliminate many of the troublesome problems encountered in P.C.E. determinations.

LITERATURE REVIEW

Pyrometric cones may be described as slender, trihedral pyramids which are prepared from blends of clays, and other oxides. The various constituents are blended in definite proportions and the end point of the pyrometric cone is reproducible, if the heating rate is reproduced. (1)

A series of compositions has been developed, which, when heated at a rate prescribed by the American Society for Testing Materials deform at definite temperatures which are 36 F° (20 C°) apart and cover a temperature range from 1085° F (585° C) to 3659° F (2015° C). [2]

Pyrometric cones are important in the ceramic industry, because they respond to both temperature and time, as well as atmosphere, not temperature alone. The materials from which cones are compounded undergo thermo-chemical reactions similar to the ceramic products being fired and, therefore, provide an accurate measure of the heat treatment received by materials during firing.[3]

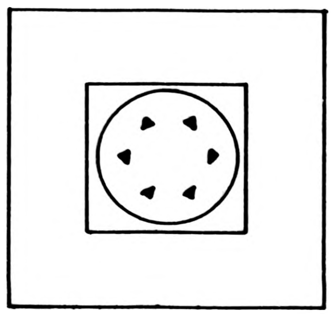
The pyrometric cone equivalent, fusion temperature of ceramic material, may be determined by preparing cones of the material and observing the heat treatment effect upon the prepared unknown cones and members of the standard series. Firing schedules, cone compositions, cone manufacture, and procedures for preparing cones from materials to be evaluated are prescribed by the American Society for Testing Materials and are presented in the handbook for Refractories.[2] The furnaces presently employed for P.C.E. determinations may be categorized into three types based on the type energy utilized to produce the necessary heat for fusion, and the manner in which the test specimens may be observed when determining the end point of the test, namely: 1) the combustion, well type furnaces, 2) the resistance, well type furnaces, and 3) the resistance, horizontal type furnaces. The design and operating characteristic of each of these furnace types are as follows:

1. <u>The Combustion, Well Type Furnaces</u> - There are two commonly used furnaces of this type, a) The Remmey Fusion Test Furnace, and b) The Denver Fire Clay (D.F.C.) P.C.E. Furnace.

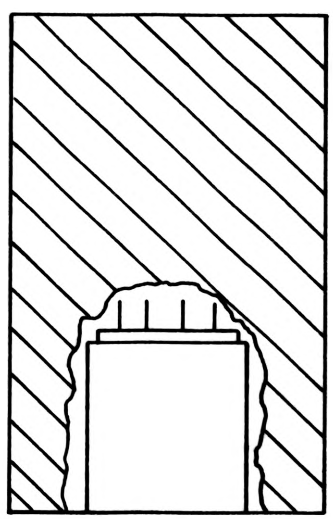
a) Remmey[4] describes "The Remmey Fusion Test Furnace" as follows: Basically, this furnace is a pot type furnace which utilizes acetylene and oxygen for combustion. The higher calorific value of the acetylene gas allows higher temperatures to be obtained, and, if a zircon or stabilized zirconia lining and pedestal is used in the furnace, a temperature of 4000° F may be attained. When ordinary fusion temperatures are required of this furnace, one troublesome problem is minimized. The higher heat content of the acetylene gas allows temperatures in the 3200° F range to be reached with relatively small volumes of combustibles as well as combustion products. Also this same property (higher heat content) will allow the desired temperature conditions with an excess of oxygen present to insure oxidizing conditions in the furnace at all times. However, the possibility of reducing conditions still exists as well as improper visibility of cones during deformation. (see Fig. 1) Another problem encountered is the proper manipulation of valves to reproduce prescribed firing schedules.

b) Cartwright and Phelps[(5)] describe a D.F.C. P.C.E. furnace. This furnace is basically a pot type furnace, which utilizes natural gas, compressed tank gas or manufactured gas in conjunction with forced air for combustion. The ultimate temperature of this furnace is greatly limited. However, if the air is enriched with oxygen, temperatures in the order of 3600° F may be reached. The volume of gases necessary to obtain this end temperature is large and the resultant turbulence created in the furnace definitely influences the cone deformation. In addition, being at the upper limit of the possible temperature condition of the combustion, any excess oxygen in the furnace would tend to cool rather than heat the furnace contents. Therefore, the usefulness of the furnace is limited, if a definite

VISIBILITY OF CONES IN A
WELL TYPE GAS FIRED FURNACE
FIG. I



CONE VISIBILITY
FROM TOP ONLY



NO VISIBILITY OF
CONES FROM THIS VIEW

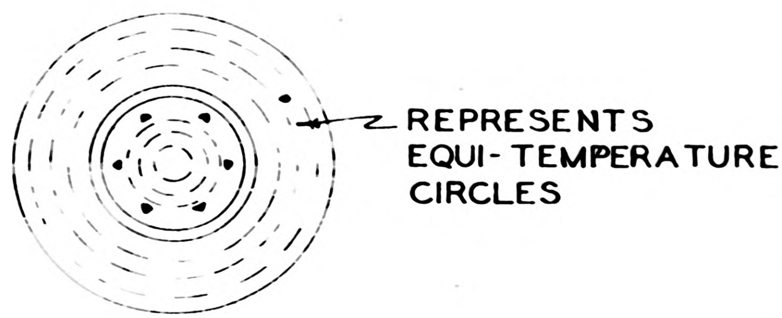
oxidizing condition is maintained. In addition to the highly turbulent gas flow, the frequent presence of reducing conditions and improper visibility of cone deformation limit the useful high temperature applications of this furnace. However, the low cost and inexpensive operation of this furnace account for its common use as a P.C.E. furnace.

A comparison of the above mentioned furnaces was made by H.F. Smalley and R.B. Sosman.[6] Their comparative testing seems to favor the Remmey furnace primarily from the standpoint of less turbulent gas flow around the test cones. The cone deformation is a result of glass formation and any directional force on the cones will influence the force of gravity in the resultant deformation of the pyrometric cone. The other primary difficulty with the D.F.C. furnace was corrected by the use of a Byrant Flomixer.

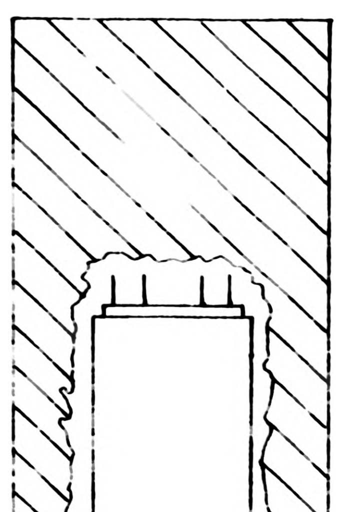
Phelps[7] discusses the work being done at Mellon Institute on P.C.E. furnaces. Various baffle configurations have been suggested and tried in an effort to improve the undesirable turbulence and also to effect more efficient heat transfer in the furnace. More efficient premixing of the gases improved reproducibility of results as well as an increase in ultimate attainable temperature. However, highly skilled operators still encountered difficulties in maintaining desired atmospheric conditions as well as maintaining proper firing schedules.

THERMAL EQUILIBRIUM AND CONE VISIBILITY IN RESISTANCE WELL TYPE FURNACE

FIG. 2



VISIBILITY OF CONES FROM TOP VIEW ONLY



2. The Resistance, Well Type Furnaces - The heating elements employed for this type furnace as well as the horizontal resistance furnace may be discussed jointly. However, the ultimate effect of the position of the heating elements relative to the cones being tested is of particular interest in this investigation. (See Fig. 2 and 3) The materials commonly used for heating elements may be classified as 1) non-oxidizing refractory metals or alloys, 2) the oxidizing metals, alloys, or carbon which must be protected with a reducing or inert atmosphere, or a vacuum. The maximum temperature, therefore, is dependent on the softening point of the particular element or alloy from which the heating element is composed, or the temperature limitations of the envelope which contains the necessary atmosphere for protection.

There are various modifications of the basic resistance, well type P.C.E. furnace; however, Arsem[8] gives a description of his furnace which is probably the most frequently referred to carbon resistance furnace. This furnace utilizes a carbon spiral for heating, with powdered coke insulation. These components are enveloped in such a manner that the envelope may be evacuated in order to protect the carbon components from oxidation. The entire system may then be immersed in water for cooling.

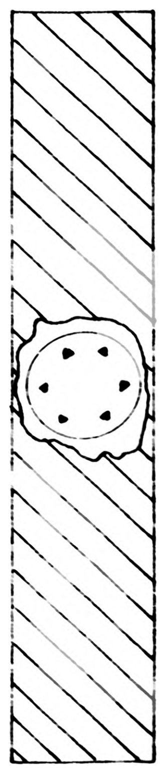
Many of the basic design features of this furnace such as the carbon resistance spiral and the total immersion feature for cooling are utilized in the design of various furnaces for fusion work other than P.C.E. determinations. The thermal conditions (See Fig. 2) obtained in this furnace, as well as other furnaces which use this basic design with different heating elements, is very desirable; however, poor visibility of test pieces (cones), and in the furnaces which use carbon heating elements, reducing atmospheres are very undesirable properties.

3. <u>The Resistance, Horizontal Type Furnace</u> - This furnace type uses, as mentioned before, heating elements similar to the resistance well type. Therefore, the use of a protective atmosphere for heating elements and the production of undesirable atmospheres by the oxidization of the elements are still problems. However, this furnace offers good visibility of cone deformation, but minimizes the desirable thermal conditions offered by the well type furnace. Increased length of the furnace cavity coupled with longer heating elements minimizes thermal gradients in the center section of the furnace chamber but does not entirely eliminate this problem. (See Fig. 3)

Norton[9] describes two types of resistance furnaces. Either could be oriented so that it could be a well type or a horizontal type furnace. Each utilizes a carbon tube for the heater and lamp black for insulation as well as to provide

THERMAL EQUILIBRIUM AND CONE VISIBILITY
IN RESISTANCE HORIZONTAL TYPE FURNACE

FIG 3



REPRESENTS EQUI-TEMPERATURE CIRCLES

CONE VISIBILITY FROM FRONT VIEW

a reducing condition in the vicinity of the heater that consequently prevents oxidation. It is necessary, in furnaces of this type, to supply a refractory inner tube in which the test cones are placed and through which a stream of air is passed, if an oxidizing condition is necessary.

A modification of the above carbon resistance furnaces is one in which induction heating techniques are used to couple the power supply with the heater.

Another modification of the resistance well type furnace is described by Lang and Geller.[10] This furnace is different because of the type heating element used. The element for this furnace is composed primarily of Thoria, a refractory oxide. At elevated temperatures Thoria has a behavior similar to that of a semi-conductor. In this application the heating element is preheated by a secondary heat source to lower the resistance of the element so that an economical and safe voltage will provide an adequate power supply. Zirconia behaves in the same manner. The cost of a furnace of this type is great and, therefore, not desirable for this application. Auxiliary equipment necessary for this furnace is not readily adaptable to other apparatus without major modifications which further increases the basic cost.

Covan and Carruthers[11] describe a modification of the Gran-Annular Electric Furnace[12] in which a rotating pedestal has been added to minimize the effects of hot spots. Slight reducing conditions and poor visibility are the primary objections to this furnace.

This paragraph will be used for additional clarification of the indicated problems. The horizontal cylindrical cavity type furnaces offer good visibility of samples but lack the desired homogeneous thermal conditions because from the outer wall to the center of a heated cylinder, the thermal gradient decreases in a circular pattern toward the center. Each circle described (using the center of the cylinder as a center of the circles) by a radius will have a constant temperature, but the circles of greater radius will have a greater temperature if the outer wall is the heat source. (See Fig. 3) This effect would cause a temperature differential from tip to base on each cone in the plaque and in every case would cause the tips of the cones to be at a higher temperature than the base or an intermediate point on the cone.

The gas fired, well type furnaces are commonly used for P.C.E. determinations up to cone 37. More expensive furnaces of this type for higher temperature work have been constructed with zirconia linings and utilize acetylene gas and oxygen to reach the desired end temperatures. Furnace construction, as well as furnace operation, is fairly expensive. The furnace atmosphere is difficult to control which not only alters the deformation of the cones but impairs the visibility as well. When accomodations are incorporated in the design of these furnaces for better visibility of samples, uniformity of heat in the firing chamber must be sacrificed. The well type, resistance furnaces in all instances sacrifice good visibility of samples which is extremely necessary when determining the end point of a particular cone. Also, the granular carbon furnace of this type develops hot spots, which causes erratic deformation of cones. The presence of the carbon increases the possibility of reducing atmospheres, which also alters the end points of cones being tested. Furnaces of this type, which use oxidation resistant refractory metal windings, produce homogeneous thermal conditions in the furnace, but again offer poor visibility of the samples as well as maximum temperature conditions which are below those desired.

Smyth, Meinken, and Wisnyi,[13] have described a vacuum, induction furnace which utilizes molybdenum or tantalum susceptors. The susceptor, fabricated from 0.005" molybdenum or tantalum sheet, was cylindrical in form, produced by joining the edges of the sheet in a double fold pressed seam. The electrical connection provided by this joining technique was reported to provide uniform heating. A two or three turn spiral of 0.025" molybdenum sheet was used as a reflector insulator, and the induction coil was made of 1/4" copper tubing. The furnace components were enclosed in a brass shell with removable top and bottom plates, which were sealed to the shell by silicone rubber gaskets. The susceptors and reflectors were supported by a zirconia pedestal. This furnace was not basically designed for P.C.E. determinations; however, it does establish the techniques for induction heating with molybdenum susceptors.

Norton[14] and Johnson[15] also describe furnaces of this general design; however, these furnaces do not lend themselves to P.C.E. determinations and are not basically constructed with this purpose in mind.

The literature review revealed problems presently encountered in P.C.E. furnaces, which the furnace designed for this problem will help to minimize or eliminate. These problems are as follows:

1. Reducing atmospheres
2. Turbulent gas flow
3. Poor visibility of test cones
4. Undesirable thermal gradients

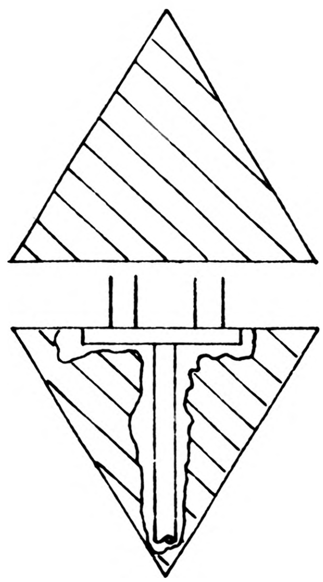
III EXPERIMENTAL

The fundamental design of the susceptor components of this furnace cause it to be unique. The susceptors for this furnace are opposed 60° cones which are fabricated from 0.005" rolled molybdenum sheet, the base or larger diameter of the cones being opposed. Theoretically, the thermal conditions of the equi-temperature circle can be produced by this arrangement because the repeated reflections of primary radiation from the 60° cones will approximate black body conditions within the susceptors. However it is only an approximation, since heat losses are introduced by the separation of the susceptors, which separation is necessary to permit observation of the pyrometric cone deformation (See Fig. 4). The importance of good cone visibility is apparent, when it is realized that the end point of pyrometric cone deformation is that instant when the cone tip touches the cone plague.

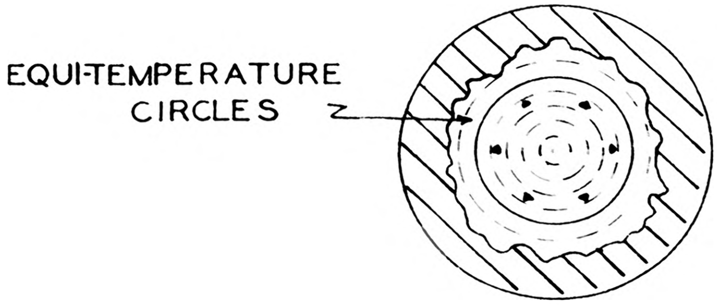
Because of the nature of the furnace it was necessary to select appropriate materials for the various components. Carbon susceptors are used in many induction furnaces; however, in this application carbon susceptors were not considered desirable because of the possibility of reducing atmospheres being produced in the immediate vicinity of the carbon. Reducing conditions in the furnace would cause erroneous deformation of the cones because of

THERMAL EQUILIBRIUM AND P.C.E. CONE VISIBILITY IN OPPOSED CONE FURNACE

FIG. 4



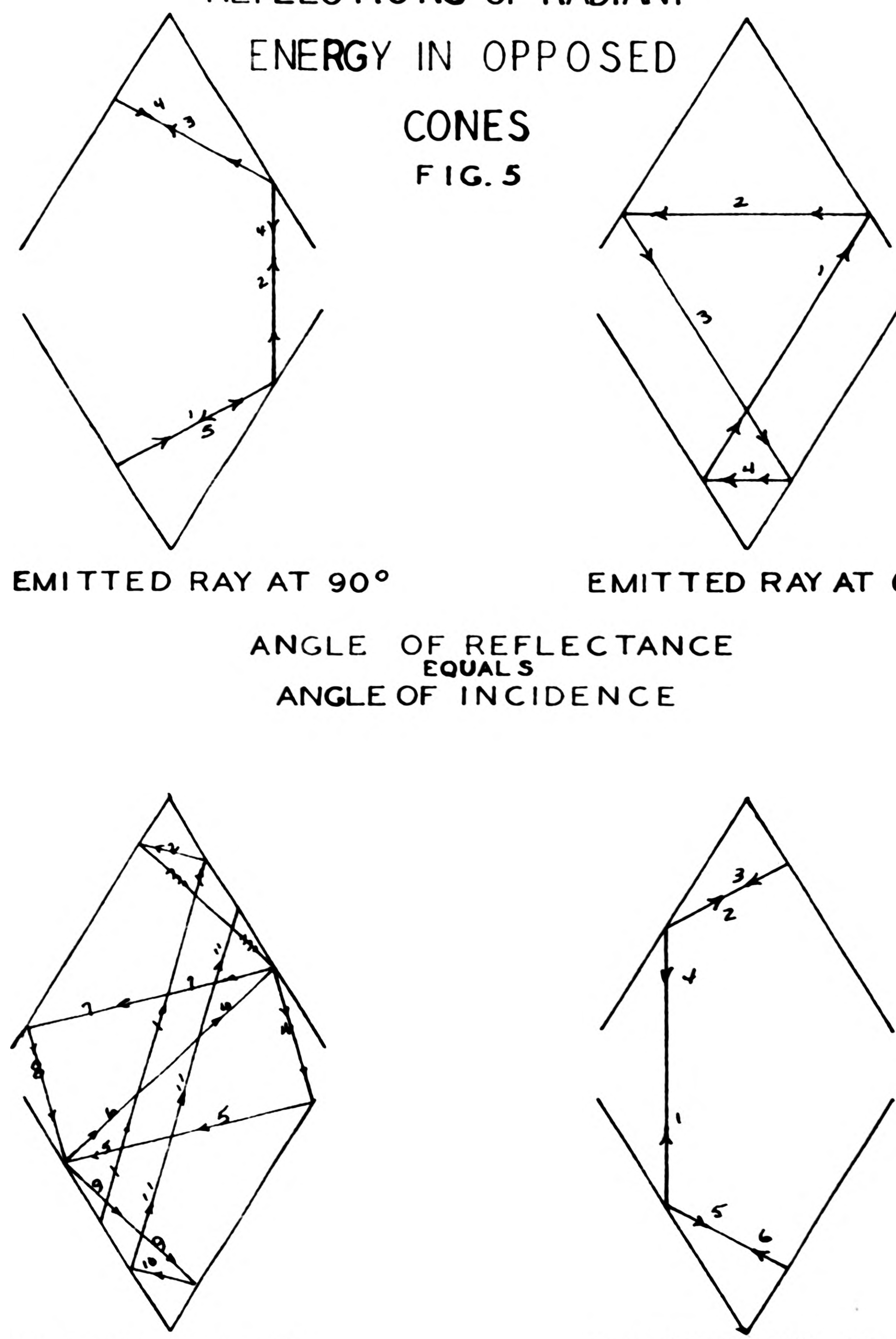
ILLUSTRATES THE EXCELLENT P.C.E CONE VISIBILITY



ILLUSTRATES THE DESIRABLE THERMAL CONDITION OFFERED BY THIS DESIGN

REFLECTIONS OF RADIANT ENERGY IN OPPOSED CONES

FIG. 5



EMITTED RAY AT 90°

EMITTED RAY AT 60°

ANGLE OF REFLECTANCE EQUALS ANGLE OF INCIDENCE

EMITTED RAY AT 75°, 45°, & 15°

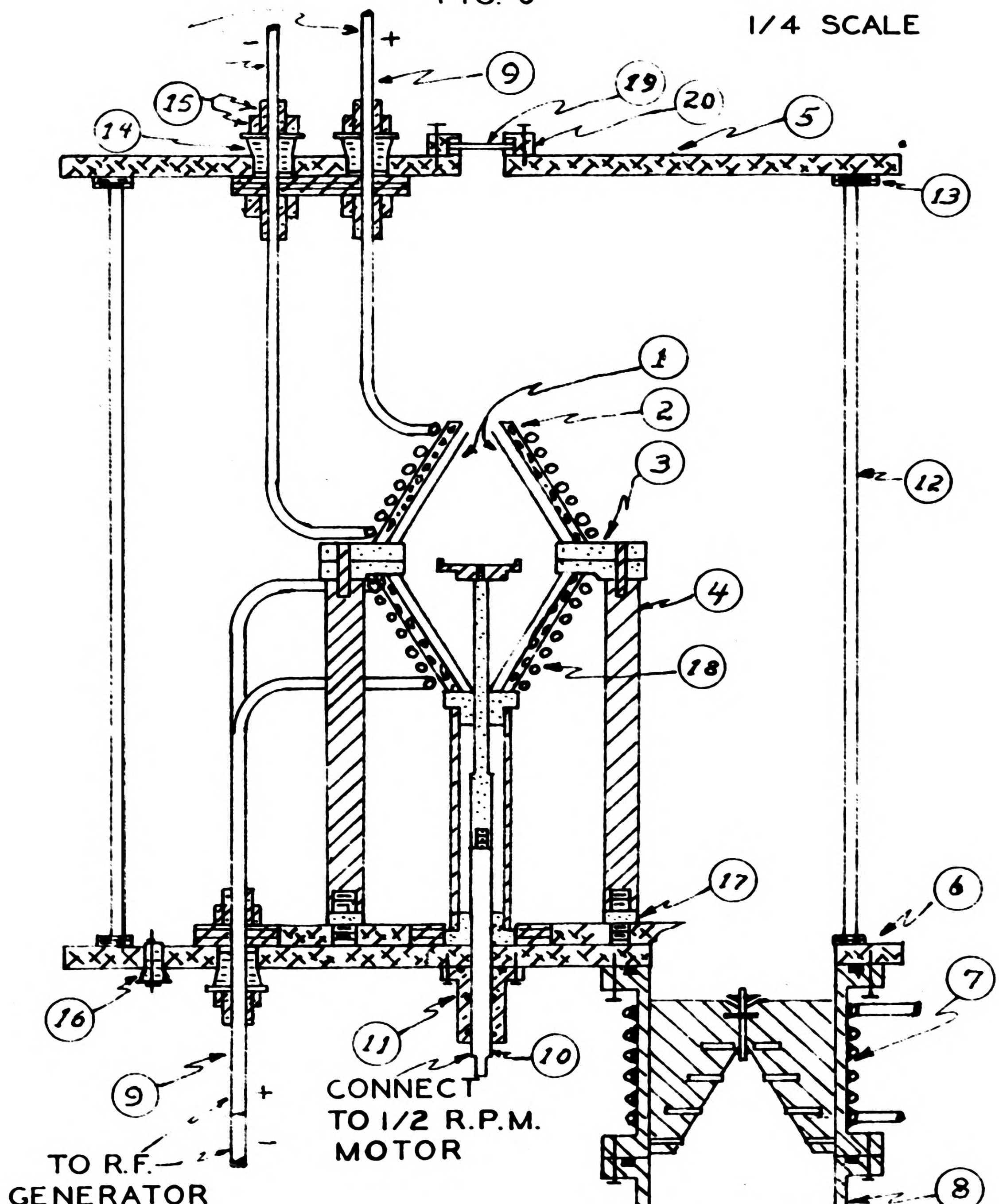
EMITTED RAY AT 30°

its influence on the various oxides present.

1. Materials - The materials, which are to be subjected to extremely high temperatures in the furnace, must be stable, have low out-gassing tendencies, and should not give rise to reducing atmospheres in the vicinity of the pyrometric cones. To minimize most efficiently these undesirable conditions, molybdenum was selected for the susceptor material, and for electrical insulating, susceptor supports, boron nitride was selected (See Fig. 6). The molybdenum was purchased from Fansteel Metallurgical Corporation and the boron nitride from the Carborundum Company. To minimize heat losses, it is necessary to envelop the susceptors in a material of high reflectivity or low emissivity. In vacuum, the predominant heat losses are from radiation; therefore, highly reflective surfaces are the most efficient means of minimizing the loss. In the first trial, continuous conical spirals, fabricated from 0.005" sheet molybdenum were used, however, the edges of the material in the regions of high flux heated more rapidly than the susceptors and were, therefore, discarded. It is possible that this behavior could have been eliminated, if molybdenum of greater thickness, thus, lower resistance, had been used. Ceramic cones of low emissivity

OPPOSED CONE VACUUM INDUCTION FURNACE

FIG. 6



DESCRIPTION OF SYMBOLS AND PARTS FOR FIGURE 6

Figure 6A

ALUMINUM

BRASS

RUBBER

CARBON

BORON NITRIDE

ALUMINA

STEEL

PYREX

1 MOLYBDENUM SUSCEPTORS

2 ALUMINA REFLECTIVE INSULATORS

3 BORON NITRIDE SEPARATORS

4 CARBON SUPPORTS

5 TOP PLATE

6 BOTTOM PLATE

7 WATER COOLED BAFFLE

8 DIFFUSION PUMP

9 INDUCTION COIL LEADS

10 STAINLESS STEEL SHAFT

11 DOUBLE "O" RING SHAFT SEAL

12 PYREX CYLINDER (ENVELOPE)

13 NEOPRENE RUBBER GASKETS

14 RUBBER STOPPER

15 THREADED SLEEVE & NUT

16 THERMOCOUPLE LEADS SEAL

17 BORON NITRIDE INSULATOR

18 INDUCTION COILS

19 POLISHED FUSED SILICA SIGHT GLASS

20 & HOLD DOWN FOR RADIATION PYROMETER

were then made and have proved more satisfactory; however, there is room for improvement. The envelope for the system should be reasonably heat resistant, non-porous, offer good visibility, and be fairly inexpensive as well as possessing low out-gassing characteristics. A pyrex cylinder with 1/4" wall thickness was chosen for this component. The top and bottom plates, as closures for the pyrex cylinder, were made from available 1/2" aluminum stock. Neoprene gaskets are used to seal the top and bottom plates to the pyrex cylinder, and Neoprene "O" rings are used for seals to the diffusion pump.

2. Vacuum Pumping System - There are two basic types of pumps for vacuum systems, the mechanical pump and the diffusion pump.

A. The Mechanical Pump - The mechanical pump is generally rated according to its free air capacity, that is the amount of gas that can be pumped through the system with atmospheric pressure on both sides of the pumping mechanism. The ultimate system pressure is dependent on the leaks present and the pumping speed. The pumping speed of the mechanical pump decreases exponentially with decreasing pressure.

B. <u>The Diffusion Pump</u> - The diffusion pump consists of a heater, a system of venturies, a confining cylinder, baffles which are used for controlled directional vapor flow, and copper cooling coils. The function of the above mentioned components are as follows:

a) The heater vaporizes a specially prepared, low vapor pressure oil.

b) The venturies accelerate the vaporized oil and the baffles cause the accelerated oil to be directed downward. The large cross-section of the long chain organic molecules (pump oil) collide with air or gas molecules and in their accelerated condition will force the air or gas molecules toward the bottom of the diffusion pump, actually compressing the air molecules in the lower portion of the containing cylinder.

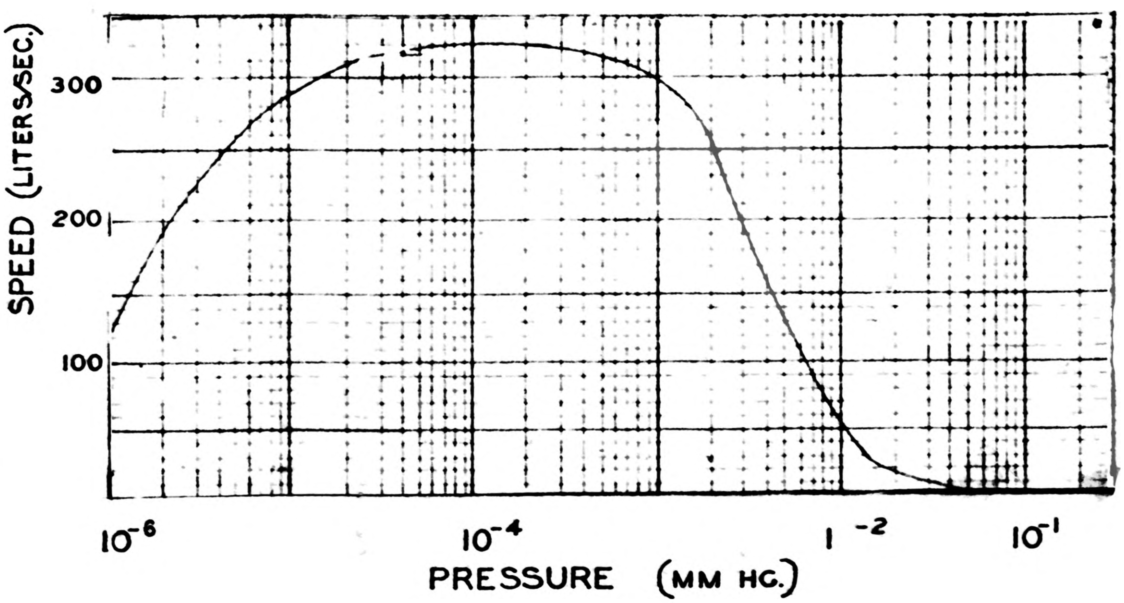
Consequently the efficiency of the diffusion pump increases with decreasing pressure and the pumping speed increases (See Fig. 7).

The combination of diffusion pump and mechanical pump creates a high pumping speed at low pressures (1 to 5 microns). The mechanical pump (fore pump) is coupled to the diffusion pump in the area where the gas molecules are compressed thereby creating a higher pressure for the fore pump to work against.

PUMPING SPEED VERSUS PRESSURE

FOR A G.E.C. MCF 300 DIFFUSION PUMP

FIG. 7



HIGH VACUUM VAPOR PUMPS, BUL. 6-1, JAN. 1958,
CONSOLIDATED ELECTRODYNAMICS, ROCHESTER, N.Y.

It is almost impossible to construct a leak free system; also the components of the furnace and system experience a certain amount of out-gassing as they are heated. It is therefore necessary and is standard practice to employ a pumping system that has a greater capacity than is actually necessary.

Formulas useful in calculating the necessary pumping capacity and speed for the system are listed in appendix 1.

In systems where extremely low pressures are an absolute necessity, a baffle may be employed to alleviate the problem of back-diffusion of oil vapor from the diffusion pump. In this system, the extremely high voltages caused ionization of the displaced oil vapors. The increase in gas volume, caused by thermal dissociation of diffusion pump oil vapor by the heated susceptors, entering the diffusion pump displaced additional vapors, which migrated into a field of R. F. current with subsequent ionization. The cascading effect of this reaction ultimately overtook the capacity of the pumping system and the vacuum was lost. This was prevalent while the pumping system was composed of a two (2) inch diffusion pump, coupled to a five (5) cu. ft. per minute mechanical fore pump to overcome this difficulty, a four (4) inch diffusion pump, baffle, and two (2) each five (5) cubic foot per minute mechanical pumps were used to replace the above. In addition, to prevent stray R. F. current from entering the diffusion pump or baffle cavity, a grounded grid was placed over the mouth of the baffle. The diffusion pump selected for this system was a Consolidated Electrodynamics Corporation type MCF 300. Two Welch mechanical pumps are used as the fore pumps.

3. Susceptors - In many references the susceptor is commonly referred to as the work-piece. The function of this component is to transform the induced R. F. current into useful heat. In this study the shape of the susceptors was conical, and the susceptors were placed in the R. F. field so that the large diameter of the cones were opposing. The angle selected for the apex was 60°. This arrangement was selected because the indicated reflections (See Fig. 5) of primary beams of radiant energy from such opposing surfaces would yield a uniform distribution of heat over any plane normal to the axis of the susceptors. When the opposing cones are separated for visibility, there will be some heat loss encountered; however, it was assumed that the extension of this thermal gradient into the interior would not be severe and the ultimate condition after repeated reflections of radiant energy would loosely approximate black body conditions.

4. Induction Heating - The use of induction heating

techniques has become important in industrial heating and extremely important as a research tool. Its flexibility and in many cases the ease of control has made it invaluable.

> According to Cable[16] (1954, pg. 11) "Induction heating is based on the principles of the alternating current transformer. When two electrical circuits are magnetically connected, an alternating current flowing in one circuit will cause a current to flow in the other; the magnitude of the current induced in the latter is a function of (1) the magnitude of the primary current, (2) the ratio of the number of turns in the two circuits, and (3) the degree to which the two circuits are magnetically interlinked."

The interlinking of the primary current with the susceptor (work piece) is relatively inefficient; therefore, the current induced is not a direct function of the ratio of turns in the primary to turns in the secondary. However, current is induced, and the heating effect is similar to that occurring in a simple resistance heater. The current induced in the susceptor can be looked upon as the summation of all the eddy currents which are set up in the metal in an alternating magnetic field. The direction of current flowing in the susceptor will be opposite to that flowing in the coils of the R.F. generator.

According to Cable[16] (1954, pg. 15) ... the ultimate heating is dependent on the following factors:

a) the primary current

b) the frequency

c) the distance between the susceptor and the coil

d) the permeability of the material

> "Since the heat that can be produced in the metal is a simple I^2R function, the total heat will be a second order function of the four values listed above, and a first order function of resistivity of the material being heated. Consequently, if this heating effect is to be made a maximum, we must use -
>
> a) high coil currents
> b) high frequencies
> c) close spacing between the coil and the work.
>
> These values must be maintained in such a relationship as to obtain the required results in the most economical manner."

The generator selected for this furnace was a vacuum-tube, high-frequency (100,000 to 600,000 cycles per second) generator, manufactured by the Scientific Electric Company of Garfield, New Jersey. The power output of this generator is 10 kilowatts.

5. <u>Insulation and Reflectors</u> - The heat source for this furnace was enveloped and a vacuum (approximately 1 micron) was maintained in the envelope. Therefore, a vacuum being a theoretically perfect insulator for conductive

heat losses, we need only be concerned with heat losses arising from radiation. The most effective means of confining radiant energy is through the use of metallic or ceramic reflectors both of which have low emissivities. The former was attempted, using continuous conical spirals of molybdenum; however, the edges and corners of the molybdenum reflectors heated more rapidly than the susceptors. These reflectors were discarded and a ceramic body composed primarily of high purity alumina was used. This produced a white body with a fairly low emissivity. Preliminary tests proved this to be satisfactory, but at the highest temperatures obtained in this study the need for a better reflector became apparent.

6. Pyrex Cylinder (Envelope) - A pyrex cylinder was chosen to envelop the furnace because of the following properties:

A. transparent nature (visibility)

B. low out-gassing characteristics

C. thermal stability at temperatures up to 400° C.

D. impervious nature

A glass possessing an extremely low absorption for radiant energy or a higher thermal stability would be more desirable but was not available.

The pyrex cylinder was purchased from the Corning Glass

Works, Corning, N.Y. The cylinder is 18" high, 16" outside diameter, and 15 1/2" inside diameter.

7. <u>Rotating Pedestal</u> - A rotating pedestal was placed in the center of the furnace. The cone plaque was placed on the pedestal and allowed to rotate (approximately 1/2 r.p.m.) to insure that each pyrometric cone would receive equivalent heat treatment.

During preliminary tests, the motor, which drove the pedestal, was included in the vacuum system by means of an auxiliary closure sealed to the base plate with a compression "O" ring. Motor and wiring troubles made this arrangement undesirable. It was replaced with a double "O" ring shaft seal which allowed the motor to be separate from the vacuum system (See Fig. 8).

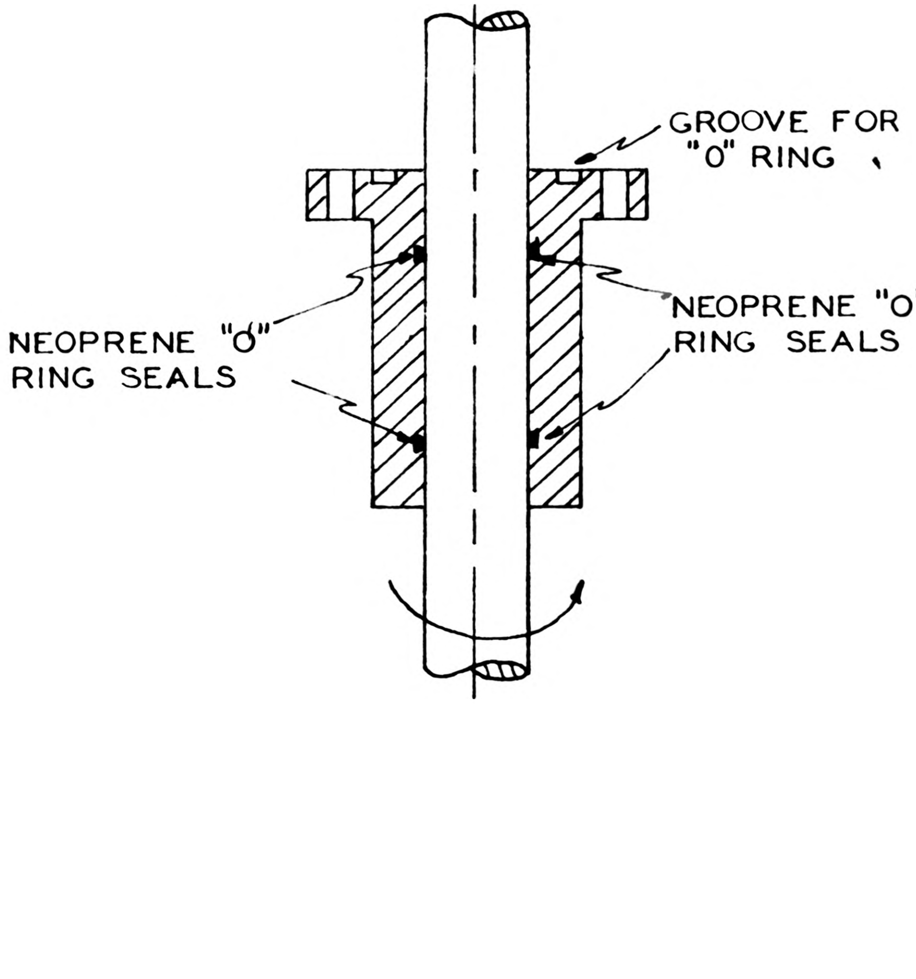
8. Vacuum Seals for Coil Leads - The vacuum seals for the coil leads and thermocouple leads were provided by using one hole rubber stoppers which are compressed by threaded sleeves attached to the coil leads. (See Fig. 9)

Figures 10, 11, and 12 are pictures of the furnace in various stages of assembly.

VACUUM SEAL FOR

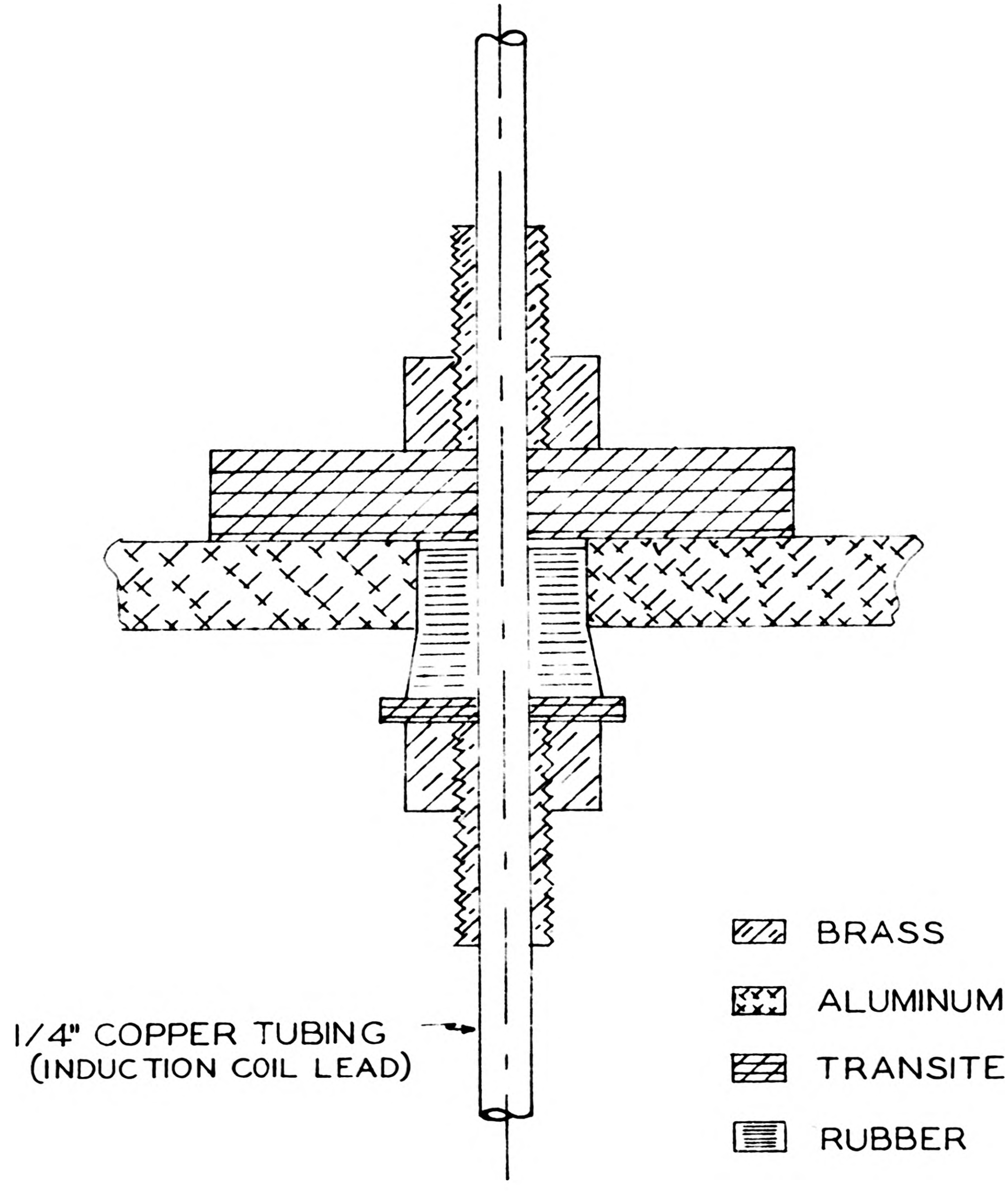
ROTATING SHAFT

FIG. 8



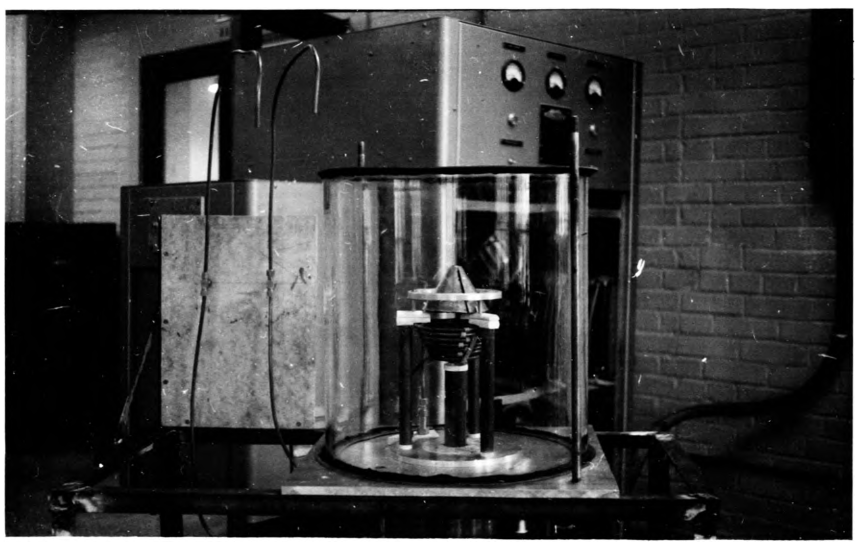
VACUUM SEAL FOR INDUCTION COIL LEADS

FIG. 9



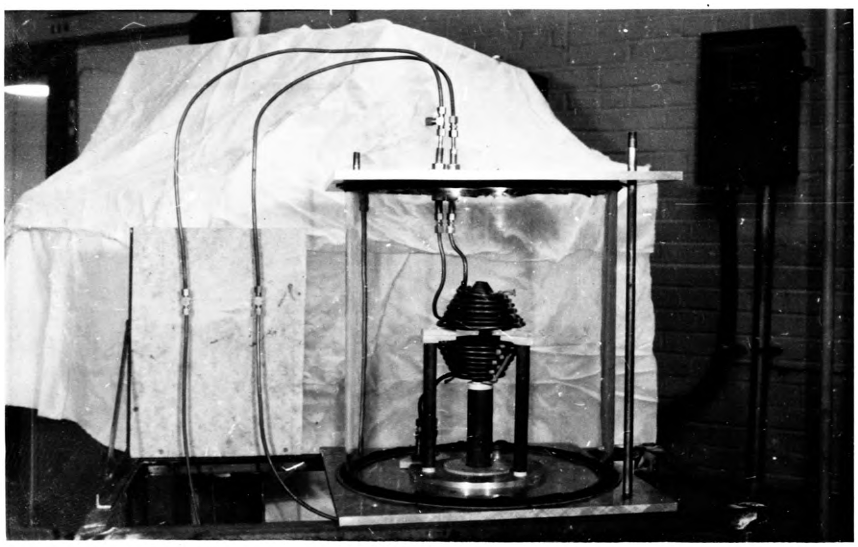
FURNACE - PARTIALLY ASSEMBLED

Figure 10



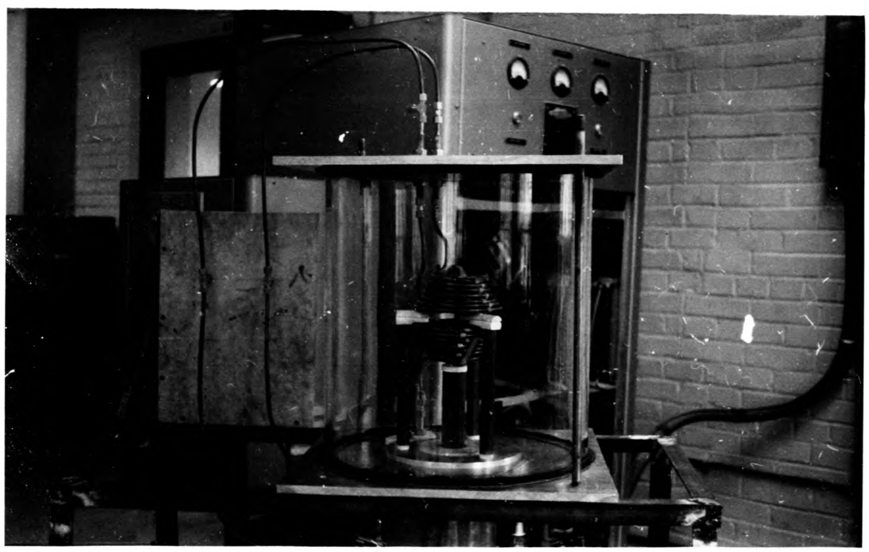
FURNACE - TOTALLY ASSEMBLED

Figure 11



FURNACE - READY FOR OPERATION

Figure 12



IV LABORATORY PROCEDURE

The apparatus was assembled according to Figure 6. After assembly, the mechanical fore pumps were turned on, and the pressure was reduced to approximately 50 to 100 microns before the diffusion pump was activated. Approximately 30 minutes were required for the diffusion pump to reach maximum efficiency. This delay can be eliminated by using a double valve system which would isolate the diffusion pump while the system is open to the atmosphere. This would eliminate the necessity for cooling the diffusion pump oil to open the system, and then reheating it for operation. The valve system would also allow the mechanical pump to be coupled to the system or the diffusion pump forearm, whichever might be necessary.

After a pressure of approximately 1 micron was attained in the system, the R.F. generator was energized. The generator used in this work had twenty oscillator tubes in a parallel circuit, and it was necessary to allow approximately 10 minutes for the cathodes of these tubes to reach ambient temperatures before operation.

When the components of the system had been open to the atmosphere for prolonged periods, it was necessary to increase the power gradually in order to heat the susceptors slowly and consequently the system. This allowed the system components to out-gas at a rate consistent with the pumping speed of the diffusion and mechanical pumps. Table I provides data indicative of the out-gassing characteristics of molybdenum at different temperatures. Pre-treatment of the molybdenum eliminates many occluded gasses; however, an additional atmosphere furnace would be necessary to perform these operations. This would be time consuming and expensive.

The first evaluation of the thermal conditions in the furnace was made with a Pt-Pt 10% Rh thermocouple attached to a post on the rotating pedestal. The pedestal was manually rotated and the temperature was determined at eight different positions (See Fig. 13) described by the rotation. At each position a 30 second soaking period was allowed for thermal equilibrium to be attained. This test is more rigid than normal operation because in normal operation all eight positions would be visited in two (2) minutes.

The second evaluation consisted of the deformation of four (4) standard pyrometric cones mounted in a cone plaque in the conventional manner.

TABLE I

OUT-GASSING CHARACTERISTICS OF MOLYBDENUM *

Brightness Temperature °C	True Temperature °C	Analysis of Gas Evolved (Percent)			
		N_2	CO	CO_2	H_2
740	790	4	75	21	
940	1010	16	42	15	27
1120	1210	81	15		4
1312	1440	76	10	6	8
1600	1760	45	24	7	24

NOTE. The above analysis are of gases evolved from a 4.9 gm. sample of molybdenum at the indicated temperature. The molybdenum was cleaned in a hot caustic solution before heating was commenced. The method of analysis was essentially that of Langmuir, as employed by Norton and Marshall (see reference).

* F.J. Norton, A.I. Marshall, "The Degassing of Metals" Trans. Inst. Min. and Met. Eng. 156, 351, (1944)

V RESULTS AND DISCUSSION

Table II and III list the pressures and temperatures obtained during an evaluation run using the 4 inch diffusion pump and the two mechanical fore pumps.

Table IV lists the temperature observed at each of the eight stations described by the rotation of the pedestal after a constant temperature condition had been attained at position one.

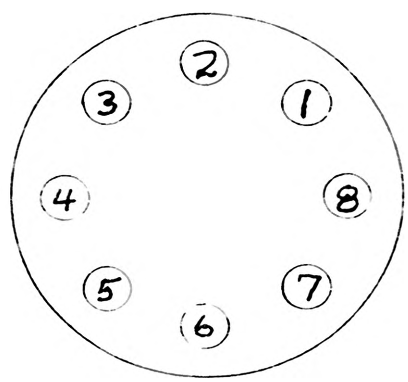
The above mentioned data may be summarized as follows:

1. At no time during the heating (firing) operation did the pressure exceed 4 microns. In previous work it was noted that after the pressure exceeds 6 to 10 microns the pumping speed was decreased because greater volumes of gasses were formed by cracking of the displaced pump oil vapors. These voluminous gasses displaced more pump oil vapors which migrated to the heated susceptors and was cracked. When this condition occurred, the vacuum was lost immediately.

2. After an equilibrium temperature was reached for position one (1) (See Fig. 13) the temperatures at the eight positions in Figure 13 were measured. A temperature difference of 4° C. was observed; the temperature range being 981° C.

TEMPERATURE MEASURING POSITIONS
FOR THERMAL
EQUILIBRIUM EVALUATION

FIG. 13



A MINIMUM OF 30 SECONDS SOAKING TIME WAS ALLOWED AT EACH POSITION to 985° C. This temperature differential should decrease at higher temperatures because the condition is caused primarily by radiation. More energy would be transferred to the components which are intended to be heated, because radiation from a source increases as a fourth power of the temperature (T^4).

3. From Table III it will be noted that at temperatures above 1100° C. on the cone plague, the ceramic reflectors did not perform satisfactorily. When temperatures in the neighborhood of 1100° C. were reached, the outer surfaces of the insulator reflector cones became dull red. This indicated that much of the radiation was being absorbed and conducted through the insulator reflector cones. This temperature condition near the R. F. induction coils, cracked the diffusion pump oils producing lower vapor pressure oils, which were condensed on the coils. This large volume of vapor in the immediate vicinity of the R. F. field was, subsequently, ionized. Also, when this quantity of energy was allowed to pass through the cones, the pyrex outer envelope was heated excessively.

4. From Table II it will be seen that the leak rate of the system is relatively high. The ultimate pressure of the mechanical pump in a leak free system should be in the 1 micron range. From the data, it will be seen that the lowest attainable pressure with the mechanical pumps was 86 microns. If the leak rate was reduced, a larger percent of the pumping system capacity would be utilized to control out-gassing of furnace components.

TYPICAL DATA FROM OPERATION

TABLE II

Time	Total Time (Min.)	Pressure in microns	Temperature (°C)	Oscillator Currents Grid (amp)	Oscillator Currents R.F. (amp)	Oscillator Currents Plate (amp)	Remarks
8:30	0	86	Rm	0	0	0	(1)
9:00	30	0	Rm	.12	16	2.5	(2)
9:02	32	0	972	.12	16	2.5	
9:06	36	0	1094	.12	16	2.5	
9:10	40	0	1140	.12	16	2.5	
9:11	41	0	1140	.12	16	2.5	(3)

(1) Only mechanical pumps operating. Started diffusion pump.

(2) Power on

(3) Short in generator power off.

TYPICAL DATA FROM OPERATION

TABLE III

Time	Total Time (Min.)	Pressure in microns	Temperature (°C)	Oscillator Currents Grid (amp)	Oscillator Currents R.F. (amp)	Oscillator Currents Plate (amp)	Remarks
12:52	0	0	Rm	0	0	0	(1)
1:03	11	1	884	.08	10	1.45	
1:10	18	1.5	936	.08	10	1.45	
1:15	21	2.5	942	.08	10	1.45	
1:17	23	2.3	942	.08	10	1.45	(2)
2:07	73	0.5	Rm	.13	17.5	2.5	(3)
2:18	84	1.5	1313	.12	16.0	2.45	(4)

(1) Power turned on. Diffusion pump on approx. 30 min.

(2) Power off.

(3) Power on.

(4) Pyrex cylinder became hot, reflectors dull red, power off.

THERMAL EQUILIBRIUM EVALUATION

TABLE IV

Position	Potentiometer reading, Actual (millivolts)	Corrected for room Temperature (millivolts)	Temperature (°C)	Remarks
1	9.21	9.36	981	A minimum of 30 seconds allowed at each position before potentiometer was read.
2	9.20	9.35	981	
3	9.22	9.37	983	
4	9.22	9.37	983	
5	9.23	9.38	984	
6	9.25	9.40	985	
7	9.20	9.35	981	
8	9.20	9.35	981	
1	9.20	9.35	981	

In the final evaluation, a cone plaque with 4 properly set standard pyrometric cones, number 5, was placed on the pedestal. The furnace was readied for operation as outlined above, and after a vacuum of 0 to 1 micron was obtained, the induction generator was placed in operation. The oscillator power input was increased until an R.F. current of 16 amperes was reached. The resultant plate and grid currents are tabulated in Table V. The temperature was allowed to increase to the limit for this particular power input. The temperature was determined by means of a Pt- Pt 10% Rh couple located on the cone plaque. The results of this test are tabulated in Table V but may be summarized as follows:

1. After the power was turned on, 15 minutes were required to reach a temperature of 1318° C.

2. At this temperature, the four number 5 Orton Standard cones were uniformly one-third the way down, and the bending or deformation was in the direction established.

3. Visual examination through a blue glass filter indicated the ceramic components within the susceptor cones were at a very uniform temperature. Due to higher temperature and low emissivity the

susceptors were more incandescent than the ceramic components, which provided a very good contrast for observing the pyrometric cones.

4. As the heating was continued various components of the furnace were subjected to a higher temperature than previously encountered. Volumous out-gassing occurred with subsequent ionization and loss of vacuum.

5. Although from all indications, the thermal conditions were very uniform, the atmosphere within the enclosure left something to be desired, in that there was a noticeable reducing condition in the furnace. It is felt that this resulted from the failure of the baffle to trap properly all oil vapors that escaped from the diffusion pump, or from the ionization by the R.F. field of various organic substances such as, high vacuum grease or Glyptol which was used in various sealing applications.

6. The temperature of the "Pyrex" envelope approached its allowable maximum temperature.

An additional test was made using helium as an inert atmosphere for protection of the molybdenum susceptor instead of the vacuum. The system was evacuated and then filled with helium. This was repeated two times and then the power was turned on. Arcing and ionization was very pronounced. Maximum temperatures obtained were in the order of 700° C. It is felt that this ionization of the helium was caused by the high voltage R. F. field.

DATA FROM FINAL EVALUATION OF FURNACE

TABLE V

Time	Total Time (Min.)	Pres-sure in microns	Temp-erature (°C)	Grid (amp)	R.F. (amp)	Plate (amp)	Remarks
10:00	0	AtMS.	25	0	0	0	(1)
11:00	60	80	25	0	0	0	(2)
11:30	90	4	25	0	0	0	
							(3)
3:00	5 hr.	0	25	.12	16	2.45	(4)
3:07	5 hr. 7 min.	2	1252	.11	15	2.4	
3:15	5 hr. 15 min.	2.5	1318	.12	15	2.4	
3:16	- - - - - -	- - - -	- - - -	- - -	- - -	- - -	(5)

(1) Mech. pump on.

(2) Diff. pump on.

(3) Power was intermittently turned on and off to out-gas system.

(4) Power on.

(5) Ionization; lost vacuum, cones uniformly 1/3 the way down. Thermal conditions very uniform.

VI CONCLUSIONS AND RECOMMENDATIONS

The following conclusions are based on the results discussed in the previous section:

1. Uniform thermal conditions exist in an area between the two susceptor cones.
2. Cone (P.C.E.) deformation caused by volumous turbulent gas flow was completely eliminated.
3. The uniform flux of the R.F. field eliminates the development of hot spots in the resistance elements (susceptors).
4. Extremely good visibility of cone deformation (P.C.E.) is possible without creating undesirable thermal gradients.
5. The use of a radiation pyrometer for temperature control is entirely feasible with this furnace, however, was not used in this project because of an equipment malfunction.
6. The ceramic reflective insulators are not as efficient as desired.
7. The reducing condition which was created by organic substance in the furnace is very undesirable and must be eliminated to permit the furnace to function properly at the higher temperatures required.

To correct this, it will be necessary to improve the leak rate, and baffling and pumping capacity of the system.

8. The "Pyrex" cylinder is not entirely satisfactory as the outer envelope because of its temperature limitations. The absorption of radiant energy is greater than anticipated and some improvement is needed.

The results and conclusions indicate that a very uniform thermal condition coupled with extremely good visibility exists in the heated zone. To improve the undesirable characteristics of the furnace, the following recommendations are made:

1. Improvement of the reflective insulators will require several trials of various substances. However, the following suggestions might prove to be adequate:

   A. Use a 2 or 3 turn conical spiral made from 0.025" or thicker molybdenum. Corners and edges could possibly be rolled or burnished to eliminate the high edge resistance. The fact that the reflective spirals are not continuous, and are perpendicular to the magnetic flux, should minimize the tendency of heating more rapidly than the susceptors.

B. An improved ceramic reflector may be provided by fabricating these components from a "bubble alumina" castable. This would yield a reflector with a relatively low emissivity as well as low thermal conductivity which might be used in conjunction with a metallic reflector which would be very desirable. Other materials from which the ceramic insulator or reflector might be made are zirconia or magnesite.

2. The back diffusion of oil vapor will be difficult to eliminate, but it is possible this behavior can be sufficiently minimized to permit operation at elevated temperatures without serious arcing or detrimental reducing atmospheres.

A. The induction coils were water-cooled and were contained within the evacuated envelope. When heat is present in the furnace, the water-cooled coils become the coldest portion of the furnace interior. This affords a surface upon which the escaped oil vapors from the diffusion pump may condense. Consequently, there is a concentration of undesirable organics in the immediate vicinity of the high frequency field. This could be readily minimized by using a woven pyrex tube to envelop the copper tubing. The outer skin of the pyrex should approximately attain the atmospheric temperature of the surrounding system, while the skin in contact with the copper will be at a lower temperature. Also, the use of an induction coil made from solid tantalum rod may serve to eliminate this problem.

B. A different means of sealing the base and top plates to the pyrex cylinder, which would minimize the use of sealing greases, would remove the vapors of this material in the R. F. field. This can be accomplished by using a stainless steel ring properly grooved to contain an "O" ring and drilled so that it may be bolted to the base plate. The ring is then attached to the pyrex cylinder with epoxy resin and cured. Pyrex and stainless steel have approximately the same thermal expansion; therefore, expansion and contraction problems will not be prevalent. This type seal will expose a minimum of organic greases to the furnace interior.

C. A grounded R.F. shield could also be placed around the seal to further protect the organic greases from the R.F. field.

D. A better baffling system would also eliminate some of the vapors escaping from the diffusion pump. In addition to the water-cooled baffle, a baffle of the bayonet type which utilizes acetone and dry ice or liquid nitrogen for cooling would definitely reduce the oil vapor problem.

3. The pyrex cylinder is a suitable envelope insofar as visibility is concerned; however, its absorption of radiant energy is great enough to create problems at higher temperatures. A first trial to eliminate this problem was not completely successful. A radiation shield was made from aluminum foil and placed inside the pyrex shell. This contained all the heat and caused unnecessary out-gassing of furnace components and created

a pumping problem. To more effectively protect the pyrex shell, which has atmospheric pressure on one side and a vacuum on the other, it would be desirable to have an additional pyrex cylinder that is not the vacuum envelope, but would have the same pressure (1 to 5 microns) on each side, that would absorb the radiation which heats the pyrex outer shell and allow the remainder to pass through both cylinders and out of the furnace.

Another solution to the problem would be the use of a water-cooled stainless steel envelope with viewing ports made from optical grade, polished, fused silica. This would be the most desirable solution because of the ease with which various connections could be made to the shell as well as the low out-gassing characteristics of stainless steel.

VII APPENDIX

Appendix 1 - The pressure required in the system for proper protection of the molybdenum susceptors is 5 microns.

System Dimensions:

Radius = 8"
Height = 18"
$V = r^2h$
$V = \frac{3.14 \times 8^2 \times 18}{1728} = 2.1$ cu. ft.
2.1 cu. ft. = 60 Liters

Formulas For Determining Diffusion Pump Size:(17)

$S = \frac{Q}{P}$
S = Speed of Pump
Q = Maximum Throughput The Pump Must Handle
P = Pressure

Assume Pump Size:

300 Liters/sec.
$P = 5 \times 10^{-3}$mm

Therefore:

$Q = 300 \times 5 \times 10^{-3} = 1500$ u Liters/sec.

This figure is the capacity the system may handle. This capacity includes the impedence of the system, the leak rate, and the out-gassing of the system components.

Another useful formula for calculating pumpdown time or the time required to reduce the system pressure from pressure P_1 to pressure P_2 is as follows:

$$t \quad \frac{2.3V}{S} \log \frac{P_1}{P_2}$$

t time required to reduce the system from pressure P_1 to pressure P_2
P_1 high pressure (starting pressure)
P_2 low pressure required
S average speed of pump between P_1 and P_2
V volume of the system

VIII BIBLIOGRAPHY

(1) Manual Of ASTM Standards On Refractory Materials, C-8, ASTM Designation: C24-56

(2) "The Properties And Uses Of Pyrometric Cones", Orton Standard Pyrometric Cones, The Edward Orton Jr. Ceramic Foundation, 3rd Ed., Columbus 1, Ohio, 1946 48 pp.

(3) R.L. Weber, Heat And Temperature Measurement, pp. 250-252, Prentice-Hall, Inc., 1950, New York, N.Y.

(4) G.B. Remmey, "The Remmey Fusion Test Furnace", J. Am. Cer. Soc., 14 (5) 358-64 (1931)

(5) V. Cartwright And S.M. Phelps, "A Portable Pyrometric Cone Equivalent Furnace", J. Am. Cer. Soc., 14 (1) 1-7 (1931)

(6) H.F. Smalley And R.B. Sosman, "A Comparison Of Gas Fired Pyrometric Cone Equivalent Furnaces", J. Am. Cer. Soc. 2 (1) 5-11 (1953)

(7) S.M. Phelps, Am. Refr. Inst. Bull. 91 (Oct. 1952) 92 (Sept. 1953)

(8) Arsem, "Description Of Arsem Furnace", J. Am. Cer. Soc., 28, 921 (1906) Trans. A. Electro. Soc. 9, 153 (1906)

(9) H.F. Norton, Refractories, 2nd Ed. Revised, McGraw-Hill Book Co., Inc., New York, N.Y. 1942 798 pp.

(10) S.M. Lang And R.F. Geller, "The Construction And Operation Of Thoria Resistor Type Furnaces", J. Am. Cer. Soc. 34 (7) 193-200 (1951)

(11) H.E. Covan And J.L. Carruthers, "A Carbon Resistance Furnace For P.C.E. Tests", Bull Am. Cer. Soc. 30 (5) 170-173 (1951)

(12) W.L. Pendergast, "An Electric Furnace For Softening Point Determinations", J. Am. Cer. Soc. 8 (5) 319-25 (1925)

(13) H.T. Smyth, R.H. Meinken, And L.G. Wisnyi, "A High Temperature Vacuum Induction Furnace", J. Am. Cer. Soc. 34 (5) 161-163 (1951)

(14) F.H. Norton, Refractories, p. 283; 3rd Ed., revised, McGraw-Hill Book Co., Inc., New York, N.Y. 1949 782 pp; Cer. Abstracts, 1950, July, p. 145a.

(15) P.P. Johnson, "Induction Furnace For High Temperature Ceramic Research", J. Am. Cer. Soc. 32 (10) 316-19 (1949).

(16) J. Wesley Cable, Induction And Dielectric Heating, Reinhold Publishing Corp. New York, N.Y. 1954 576 pp.

(17) High Vacuum Vapor Pumps, Bulletin 6-1, Jan. 1958, Consolidated Electrodynamics, Rochester, N.Y.

IX VITA

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